

FORM PTO 1300 (REV 11-2000)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER DNAG 222 - PFF/JRC	
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371				U.S. APPLICATION NO. 09/03696 (37 CFR 1.5)	
INTERNATIONAL APPLICATION NO. PCT/EP00/03696		INTERNATIONAL FILING DATE 26 April 2000		PRIORITY DATE CLAIMED 27 April 1999	
TITLE OF INVENTION <b>GRANULATED GAS CHARGES</b>					
APPLICANT(S) FOR DO/EO/US <b>DYNAMIT NOBEL GMBH EXPLOSIVSTOFF- UND SYSTEMTECHNIK</b>					
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:					
<p>1. <input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</p> <p>2. <input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing 35 U.S.C. 371</p> <p>3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371 (f)). The submission must include items (5), (6), (9) and (21) indicated below.</p> <p>4. <input type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (PCT Article 31).</p> <p>5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371 (c)(2))</p> <p>a. <input checked="" type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau).</p> <p>b. <input checked="" type="checkbox"/> has been communicated by the International Bureau.</p> <p>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</p> <p>6. <input type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371 (c)(2)).</p> <p>a. <input type="checkbox"/> is attached hereto.</p> <p>b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4).</p> <p>7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))</p> <p>a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau).</p> <p>b. <input type="checkbox"/> have been communicated by the International Bureau.</p> <p>c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</p> <p>d. <input type="checkbox"/> have not been made and will not be made.</p> <p>8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).</p> <p>9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).</p> <p>10. <input type="checkbox"/> An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).</p> <p><b>Items 11 to 20 below concern document(s) or information included:</b></p> <p>11. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</p> <p>12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</p> <p>13. <input type="checkbox"/> A FIRST preliminary amendment.</p> <p>14. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment.</p> <p>15. <input type="checkbox"/> A substitute specification.</p> <p>16. <input type="checkbox"/> A change of power of attorney and/or address letter.</p> <p>17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.</p> <p>18. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4).</p> <p>19. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).</p> <p>20. <input checked="" type="checkbox"/> Other items or information: PCT/ISA/210</p>					

U.S. APPLICATION NO. (if known) <b>10/030007</b>		INTERNATIONAL APPLICATION NO. <b>PCT/EP00/03696</b>		ATTORNEY'S DOCKET NUMBER <b>DNAG 222 - PFF/JRC</b>																																													
17. <input checked="" type="checkbox"/> The following fees are submitted:				<b>CALCULATIONS PTO USE ONLY</b>																																													
<b>BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):</b> <input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... \$1040.00 <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... \$890.00 <input checked="" type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$740.00 <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$710.00 <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$100.00  <b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b> Surcharge of \$ <u>130.00</u> for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492 (e)).				\$ 740.00																																													
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a. <input type="checkbox"/> A check in the amount of \$ _____ to cover the above fees is enclosed. b. <input checked="" type="checkbox"/> Please charge my Deposit Account No. <u>500624</u> in the amount of \$ <u>1000.00</u> to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required or credit any overpayment to my Deposit Account No. <u>50-0624</u> . A duplicate copy of this sheet is enclosed.																																																	
<b>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.</b>																																																	
SEND ALL CORRESPONDENCE TO: James R. Crawford FULBRIGHT & JAWORSKI L.L.P. 666 Fifth Avenue New York, New York 10103 (212) 318-3148 Customer No. 24972																																																	
				SIGNATURE: <u>James R. Crawford</u> NAME: <u>James R. Crawford</u> 39,155																																													

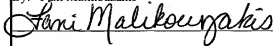
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531 Rec'd PCT/F 25 OCT 2001

## Certificate of Express Mail

This mail is being sent by Express Mail No. EL 829643533 US  
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Washington, D.C. 20231 Box PCT  
On October 25, 2001

By: Fani Malikouzakis

A handwritten signature in cursive script, reading "Fani Malikouzakis", is written over a horizontal line.

10030007-112901

DNAG 222 - PFF/JRC**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Applicants : Ulrich BLEY et al.  
Serial No. : To Be Assigned  
Filing Date : October 25, 2001 (based on PCT/EP00/03696)  
For : GRANULATED GAS CHARGES

November 28, 2001

Hon. Commissioner of Patents  
and Trademarks  
Washington, D.C. 20231

**PRELIMINARY AMENDMENT**

SIR:

Prior to prosecution, please amend the above-identified patent application as follows:

**IN THE CLAIMS:**

Cancel claims 1-10 without prejudice and substitute the following claims:

11. Granulated gas charges having combustion vapors that are free of nitrogen oxide and are deficient in carbon monoxide, comprising
- a) 5 to 50% by weight of a binding agent;
  - b) oxidizing agents in a quantity 0 to 90% by weight of an oxidizing agent; and
  - c) 0 to 75% by weight of an organic fuel.
12. Gas charges according to claim 11, wherein the binding agent is at least one binder selected from the group consisting of cellulose acetate, cellulose acetobutyrate, cellulose triacetate, nitrocellulose and polyvinyl butyral.

13. Gas charges according to claim 11, wherein the oxidizing agent is at least one oxidizing agent selected from the group consisting of from alkali perchlorates, alkaline earth metal, perchlorates, zinc peroxide, iron oxides, cerium dioxide, copper oxide, manganese dioxide and permanganates.
14. Gas charges according to claim 11, wherein the oxidizing agent is potassium perchlorate.
15. Gas charges according to claim 11, wherein the organic fuels are acids selected from the group consisting of terphthalic acid, fumaric acid and ascorbic acid.
16. Gas charges according to claim 11, wherein the gas charges further comprise at least one aggregate.
17. Gas charges according to claim 16, wherein said aggregate is a combustion moderator.
18. Gas charges according to claim 16, wherein the aggregate is selected from the group consisting of aluminum oxide, zinc oxide, alkali metal silicates, alkaline earth metal silicates, mineral clays, cement, gypsum, alkali metal carbonates alkaline earth metal carbonates, alkali metal oxalates, alkaline earth metal oxalates, and oxalic acid.
19. Gas charges according to claim 11, wherein the gas charges further comprise a coating additives selected from the group consisting of graphite, water glass, nitrates of alkali metal, perchlorates of alkali metals, nitrates of alkaline earth metals and perchlorates of alkaline earth metals.
20. Gas charges according to claim 11, in the form of a ball powder.

21. A motor-vehicle safety system comprising the gas charges of claim 1.
22. The motor vehicle safety system of claim 21, wherein said system is a belt tightening or an air bag.
23. An industrial cartridge for gas generation comprising the gas charges of claim 11.

**REMARKS**

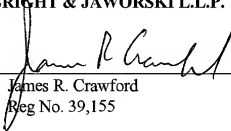
The purpose of this amendment is to conform the claims to standard U.S. practice.

Applicants believe no fee is required for filing this amendment. However, the Commissioner is hereby authorized to deduct any fee due, or credit any overpayment to Deposit Account No. 50-0624.

Respectfully submitted,

**FULBRIGHT & JAWORSKI L.L.P.**

By

  
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PTO/PCT Rec'd 28 NOV 2001

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Granulated gas charges

The subject-matter of the invention relates to granulated gas charges and also to the use thereof.

The current process for the production of gas charges, for example for motor-vehicle safety, consists in pressing the raw materials to form tablets. The desired combustion processes can be achieved with these tablets. The disadvantages of this technology lie in the cost-intensive production of the tablets and the high pressures that are required in order to attain firmness when pressing, in particular with regard to safety. Usually, the raw materials are mixed together and pressed in the dry state or, if applicable, with small proportions of pressing aids.

In comparison with this, the object of the present invention consists in making novel granulated gas charges available.

The object of the invention mentioned above is achieved by means of granulated gas charges that have combustion vapours that are free of nitrogen oxide and are deficient in carbon monoxide and which contain

- a) binding agents in a quantity of 5 to 50 % by weight,
- b) oxidizing agents in a quantity of 0 to 90 % by weight, and/or
- c) organic, in particular nitrogen-free, fuels in a quantity of 0 to 75 % by weight.

The subject-matter of the present invention relates in particular to combinations of binding agents, metal salts of oxidizing acids and/or organic, preferably nitrogen-free, fuels for use, for example, in gas

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generators as pyrotechnic mixtures for the generation of gases. The gas charges are obtained as a result of granulation of the components from a solvent-containing suspension of the components by means of the addition of water.

The gas charges that are defined in accordance with the invention are distinguished by combustion vapours which are free of nitrogen oxide and have clearly reduced amounts of carbon monoxide and by a high level of thermal stability in comparison with usual materials produced on a nitrocellulose base as the sole energy carrier. Furthermore, they are distinguished by the simple production process and the control of the combustion speed connected therewith by way of grain size, aggregates and coating compositions. The gas charges that are defined in accordance with the invention are preferably based on mixtures of nitrogen-free components as energy carriers and binders with proportions by weight of 5 to 50 % by weight, such as cellulose acetate, cellulose acetobutyrate, cellulose triacetate, nitrocellulose (here a subordinate quantity functioning as a binding agent) and polyvinyl butyral.

Oxidising agents that can be used are perchlorates, for example of the alkali and alkaline earth metals, zinc peroxide, iron oxides, cerium dioxide, copper oxide, permanganates, tin dioxide and manganese dioxide. Potassium perchlorate and zinc peroxide in proportions by weight of 0 to 90 % by weight are preferably used.

Organic nitrogen-free fuels, such as, for example terephthalic acid, fumaric acid and/or ascorbic acid, can be added in proportions by weight of 0 to 75 % by weight. Furthermore, aluminium oxide, zinc oxide, silicates of the alkali and alkaline earth metals,



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clays of differing compositions, cement, gypsum, carbonates of the alkali and alkaline earth metals, oxalic acid, for example oxalates of the alkali and alkaline earth metals, can be used as aggregates to moderate combustion.

Substances such as graphite, water glass, nitrates and perchlorates of the alkali and alkaline earth elements are suitable as coating additives.

In a preferred embodiment, the gas charges that are defined in accordance with the invention are produced by granulation to form a kind of "ball powder". By a "ball powder" is usually understood a propellant charge powder that consists of spherical powder elements and which is usually produced according to a special process developed by Olin Mathieson, USA. A high-percentage nitrocellulose solution in a solvent that cannot be mixed with water, for example methyl or ethyl acetate, is dispersed in water whilst stirring carefully in such a way that floating spheres are formed. By heating below the boiling point of the solvent, progressive reduction in the strength of solvents and thus hardening of the floating spheres is achieved.

Since the spherical form is not favourable (is particularly degressive) in terms of interior ballistics, usually far-reaching surface treatment is effected in order to surround a core that burns comparatively quickly with a shell that burns more slowly. The process for the production of the gas charges in accordance with the invention is thus distinguished by ease of handling and a high level of safety, since here operations are almost exclusively carried out with components that are moist with solvent

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and/or water.

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5 The binding agents that are defined in accordance with  
the invention are dissolved in a suitable solvent (for  
example methyl acetate) and, after dissolution of the  
binder, the oxidizing agent and the aggregates are  
added thereto. In order to precipitate the granulated  
material, water is added whilst stirring. Water-  
soluble components can then be used after previous  
10 suitable coating or by encapsulation. It is possible  
to work with correspondingly saturated solutions using  
residual solubility that still exists. It is possible  
to control the geometry, grain-size distribution and  
bulk density of the granulated material as a function  
15 of the quantity of solvent, the speed with which the  
water is added dropwise and the speed of stirring.  
After drying the granulated material, the handling  
safety and combustion behaviour can be modified in a  
manner known, per se, by means of coating additions.  
20 The coatings can either be deposited by applying the  
dry substances or by spraying in accordance with  
methods known per se.

25 Granulated materials for the purposes of the present  
invention describe the term derived from small grains  
for accumulations of small granulated grains. A  
granulated grain is thus an asymmetrical agglomerate  
consisting of powder particles (whole crystals, crystal  
fragments or particles). In contrast with the pellet,  
30 but like an agglomerate, it has no harmonic geometric  
form; the form of a sphere, a small bar, a cylinder and  
so on is only approximate and is only hinted at. The  
surface as a rule is uneven and jagged, the mass in  
many cases being more or less porous.

35 An important criterion of the gas charges in accordance

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with the invention relates to the combustibility of the spherical powder. Possible combinations of the constituents a), b) and/or c) result in compositions which cannot, however, be termed combustible ball powder for the purposes of the present invention. For the purposes of the present invention, a combustible ball powder, and thus a ball powder that is in accordance with the invention, is denoted by such a process in which the powder continues to burn after ignition even if the ignition source is removed.

The gas charges that are defined in accordance with the invention are suitable in particular for use in motor-vehicle systems, such as, for example, belt-tighteners or air bags, and industrial cartridges for gas-generation, for example in bolt-driving equipment. The gas charges that are defined in accordance with the invention are distinguished by non-poisonous combustion vapours and combustion residues.

#### Exemplifying embodiments:

The following three examples show how the gas charges in accordance with the invention behave with regard to combustion fumes and thermal stability.

#### Examples:

The composition (% by weight) and the characteristic data in terms of safety techniques of the mixtures of Examples 1 to 3 are indicated in Table 1. The specified components for the mixtures in accordance with the invention were weighed out in the weight ratios indicated and added to the binding agents dissolved in methylacetate. Subsequently, water was added whilst stirring and the granulated material that

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was formed was filtered off. After drying, the granulated material was sieved and could undergo surface-treatment. The sensitivity to friction and impacts was measured in accordance with methods of the Bundesanstalt für Materialforschung und -prüfung (BAM), which are also described, for example, in J. Köhler, R. Meyer, Explosionsstoffe, 8th edition 1995, published by VCH Verlagsgesellschaft Weinheim. The detonation point was determined by means of thermal gravimetric analysis (Mettler) and the heat of explosion was measured with a calorimeter ex EKA.

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Table 1: Overview of the mixture of Examples 1 to 3

	Example 1	Example 2	Example 3
Components in weight %			
Cellulose acetate	25	12	20
Cellulose acetobutyrate		10	
Potassium perchlorate	75	73	57
Calcium carbonate		5	
Aluminium oxide			23
Sensitivity to friction	360 N	360 N	360 N
Sensitivity to impact	10 J	10 J	15 J
Detonation Point	340°C	330°C	340°C
Heat of explosion	4300 J/g	4080 J/g	3500 J/g
Weight loss after 240 hours at 145°C	0.5 %	0.04 %	

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In order to determine the combustion properties, combustion tests were carried out in a closed 25 ml high-quality steel pressure bomb. For this purpose, the combustion tests were carried out with a 3 g weighed sample of the mixtures of the examples, with ignition being effected by means of an incandescent filament and 0.2 g of an ignition mixture consisting of boron/potassium nitrate and the pressure-time curve being plotted by means of a piezoelectric measuring system. A compilation of the pressure-rise times ( $\Delta t$ ) is given in Table 2, with the percentages relating to the pressure maximum. The composition of the combustion vapours was determined with the aid of a thermodynamic computing program (ICT Code) and is presented in Table 2.

	Example 1	Example 2	Example 3
Pressure bomb results			
Weighed sample [g]	3	3	3
Pmax [bar]	850	691	506
$\Delta t_{(20-80 \text{ s})}$ [ms]	2.6	2.7	6.1
$\Delta t_{(25-75 \text{ s})}$ [ms]	1.1	1.2	2.5
Gas composition			
Carbon dioxide [Vol %]	56	57	57
Water [Vol %]	43	42	42
Nitrogen monoxide [Vol %]	0	0	0
Carbon monoxide [Vol %]	<0.0001	<0.0001	<0.0001

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Claims

1. Granulated gas charges having combustion vapours that are free of nitrogen oxide and are deficient in carbon monoxide, containing

- a) binding agents in a quantity of 5 to 50 % by weight,
- b) oxidizing agents in a quantity of 0 to 90 % by weight, and/or
- c) organic, in particular nitrogen-free, fuels in a quantity of 0 to 75 % by weight.

2. Gas charges according to claim 1, characterized in that the binding agent is selected from cellulose acetate, cellulose acetobutyrate, cellulose triacetate, nitrocellulose and/or polyvinyl butyral.

3. Gas charges according to claim 1, characterized in that the oxidizing agent is selected from perchlorates of alkali and/or alkaline earth metals, zinc peroxide, iron oxides, cerium dioxide, copper oxide, manganese dioxide and/or permanganates.

4. Gas charges according to claim 3, characterized in that the perchlorate is potassium perchlorate.

5. Gas charges according to claim 1, characterized in that the organic fuels comprise terephthalic acid, fumaric acid and/or ascorbic acid.

6. Gas charges according to one of claims 1 to 5, characterized in that the gas charges comprise aggregates, in particular combustion-moderators.

7. Gas charges according to claim 6, characterized in that the aggregates comprise aluminium oxide, zinc

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oxide, alkali metal and/or alkaline earth metal  
silicates, mineral clays, cement, gypsum, alkali metal  
and/or alkaline earth metal carbonates, alkali metal  
and/or alkaline earth metal oxalates, and also oxalic  
acid.

8. Gas charges according to one of claims 1 to 7,  
characterized in that the gas charges comprise coating  
additives, in particular graphite, water glass and also  
nitrates and/or perchlorates of alkali metals and/or  
alkaline earth metals.

9. Gas charges according to one of claims 1 to 8 in  
the form of a ball powder.

10. Use of the gas charges according to one of claims  
1 to 9 for the production of motor-vehicle safety  
systems, in particular belt-tighteners or air bags,  
industrial cartridges for gas-generation, in particular  
for bolt-firing equipment.



**DECLARATION/POWER OF ATTORNEY**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

**GRANULATED GAS CHARGES**

the specification of which:

( ) is attached hereto.

( X ) was filed on October 25, 2001 as U.S. Serial No.  
based on PCT/EP00/03696 - International Filing Date: 26 April 2000

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

<u>199 19 065 8</u>	<u>Germany</u>	<u>27 April 1999</u>	<u>Yes (X) No ( )</u>
(Number)	(Country)	(Day/Month/Year Filed)	Priority Claimed

**U.S. Priority Applications**

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of the application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Section 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, Section §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

<u>PCT/EP00/03696</u>	<u>April 26, 2000</u>	<u>Pending</u>
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DNAG 222

(Applic. Serial No.) (Filing Date) (Status patented/pending/abandoned)

Power of Attorney

I hereby appoint the following attorneys to prosecute this application and transact all business in the Patent and Trademark Office connected therewith: John E. Lynch, Reg. No. 20,940; Peter F. Felfe, Reg. No. 20,297; Norman D. Hanson, Reg. No. 30,946; John A. Baucr, Reg. No. 32,554; James Zubok, Reg. No. 38,671; James R. Crawford, Reg. No. 39,155; Andrew Im, Reg. No. 40,657 and David Rubin Reg. No. 40,314, my attorneys with full power of substitution and revocation. Address all telephone calls to James R. Crawford, (212) 318-3148. Address all correspondence to James R. Crawford, Esq. at

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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